

RECRYSTALLIZATION; PURIFICATION of SOLID ORGANIC COMPOUNDS

REF: "Aldrich", "the CRC", "the Merck", or "Lange's"
The Organic Chem Lab Survival Manual, 7th ed., J. Zubrick, Wiley, Hoboken, NJ

EQUIPMENT: Mel-Temp apparatus & m.p. tubes
equipment in student lab drawers
Thermowell heating mantle & controller
Buchner funnel & filter flask
containers for ice baths

MATERIALS: acetanilide, impure
decolorizing / activated charcoal (*this will not be needed if the impurity is not colorful*)
filter paper (to be fluted; for gravity filtration)
filter paper (for Buchner funnels)
ice

INTRODUCTION

Most of the time, an organic compound that has been synthesized or otherwise obtained will also contain undesirable impurities. This experiment will illustrate one common technique for "cleaning up" impure samples.

Often a solid organic compound can be separated from other solid impurities by a technique called recrystallization. The Big Idea is that a suitable solvent will dissolve the desired product and the impurities, but by taking advantage of differences in concentrations and solubilities, we can precipitate the pure compound out of solution while leaving the "undesirables" in solution. (The solubility differences can especially be used to our advantage by changing temperatures.) Once the purified substance is precipitated, we simply filter out our desired product.

Your task today will be to purify your acetanilide; after your product dries for one week, you'll also determine the melting point and percent recovery.

SAFETY

Boiling water and steam can cause severe burns. Be careful handling vessels that contain hot water. As always, wear your safety goggles at all times when anyone in the class is doing experimental work.

Review Zubrick, chapter 1. If you have any questions about lab safety, ask your instructor before proceeding with any experiment.

PROCEDURE

1. Make a neat, legible list of properties for acetanilide; this should include: (ref. Zubrick ch. 3)
 - a. systematic (IUPAC) name
 - b. molecular & structural formulas
 - c. melting point
 - d. solubility information
 - e. safety and toxicity information
 - f. typical use and application for this compound

2. Determine the melting point (ref. Zubrick ch. 12) of your impure acetanilide. Discard your m.p. tube(s) in the waste jar (or beaker) in the fume hood.

3. Dissolve the Impure Acetanilide
 - a. Take a brief look at Zubrick, chapters 4, 18, 19 & 23 so that you're familiar with the contents of these chapters. You'll be referring to them for this part of the experiment.
 - b. Weigh out a 1.5 gram sample of your impure acetanilide; place this in a 100-mL round bottom flask (ref. Zubrick ch. 4). Record this mass to the appropriate precision.
 - c. Assemble a Standard Reflux setup (ref. Zubrick ch. 23 *plus* 4, 18 & 19).
 - d. Add 35 mL of deionized water to the flask through the top of the condenser.
 - e. Bring the water to a boil by heating with a Thermowell heating mantle and power controller (ref. Zubrick ch. 18).
 - f. Adjust the mantle temperature so that the water refluxes steadily.
 - g. Continue to heat until no more solid appears to dissolve.
 - h. Remove the heat source, allow the flask to cool a few minutes.
 - i. Remove the condenser temporarily, and add a small amount (~0.2 g) of activated charcoal (ref. Zubrick ch. 13) to the contents of the flask.
 - j. Replace the condenser and heat the solution at reflux for an additional 5 minutes.

4. Filter the Mixture Solution
 - a. Meanwhile, set up a gravity filtration apparatus using fluted filter paper (ref. Zubrick ch. 13); use a 125-mL (or similarly-sized) Erlenmeyer flask as the receiver.
 - b. Pour 15-20 mL of boiling water through the funnel to warm it and to wet the filter paper; discard this water.
 - c. Remove the condenser and, using the clamp as a handle, filter the hot acetanilide solution as quickly as possible. If particles of charcoal pass through the filter paper, return the filtrate to the round-bottomed flask, heat the solution to boiling, and filter it again through the same piece of filter paper.

5. Recrystallize & Recover the Purified Acetanilide

- a. Place the Erlenmeyer flask in an ice bath (ref. Zubrick ch. 20) to complete the crystallization.
- b. Meanwhile, assemble a vacuum filtration apparatus using a filter flask and Buchner funnel (ref. Zubrick ch. 13).
- c. Place a piece of filter paper into the Buchner funnel, and pour 15-20 mL of cold water through the filter paper. Discard this water.
- d. When crystallization is complete, collect the crystals by vacuum filtration (ref. Zubrick ch. 13).
- e. Store your recovered crystals in your lab drawer to dry until next week, when you will determine the yield and melting point.

6. Answer as many of the questions on pages 4-5 as possible.

One week later...

7. Determine the m.p. (ref. Zubrick ch. 12) of your purified acetanilide.
8. Weigh your dry, purified product (ref. Zubrick ch. 13), and calculate the percent recovery (ref. Zubrick ch. 2) of your acetanilide.
9. Place your acetanilide in the beaker labeled "student purified acetanilide".
10. Complete pages 4-5; attach your sheet with the properties of acetanilide (see PROCEDURE, step 1), and turn it in to your instructor **ON OR BEFORE THE DUE DATE**.

NAME: _____

DATA, CONCLUSIONS & QUESTIONS

1. melting "point" of impure acetanilide mixture _____
2. Initial mass of impure acetanilide..... _____
3. mass of purified acetanilide recovered _____
4. percent recovery..... _____
5. melting "point" of purified acetanilide mixture _____
6. What is the biggest safety hazard in this experiment?

7. What is the difference between percent recovery and percent yield?

8. What is the difference between a condenser and distilling column?

9. Melting "point" is a misleading term, especially for students. Why?

10. What happens to the impurities in a recrystallization? In other words, how are soluble impurities removed during a recrystallization?
11. Why is water a good solvent for the recrystallization of acetanilide?
12. How did the melting point of your acetanilide change after recrystallization? Why could you test the purity of your acetanilide by mixing it with a known, pure sample of acetanilide and re-checking the m.p.?
13. Why must the funnel be heated before the hot acetanilide solution is filtered?
14. What purpose does adding finely ground charcoal during recrystallization serve?